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SOUTHWEST RESEARCH INSTITUTE
San Antonio, Texas 78228

Contract No. N00156-69-C-0856; First, Second and Third Quarterly Technical Progress Report, SwRI Project No. 15-2474, Phase II, "Development of Nondestructive Testing Techniques for Detecting Stress in Brittle Materials"

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12 Aug 1970

SOUTHWEST RESEARCH INSTITUTE

8500 CULEBRA ROAD SAN ANTONIO, TEXAS 78228

3 October 1969

Supply Officer
Naval Air Engineering Center
Philadelphia, Pennsylvania 19112

Attention: AS-63 for MAM

Subject: Contract No. N00156-69-C-0856 First Quarterly Technical Progress Report, SwRI Project No. 15-2474, Phase II, "Development of Nondestructive Testing Techniques for Detecting Stress in Brittle Materials"

Dear Sir:

During the past quarter of the subject project attention has been concentrated on putting into operation the magneto-optic domain observation apparatus which was developed in Phase I of the project. This effort has culminated in very satisfactory results in the case of domains in thin films of iron deposited on glass substrates. These specimens were used initially because (1) the domains in them are large in size and hence comparitively easy to observe under low magnification, and (2) the quality of the reflecting surface of the film is high, hence difficulties due to nonspecular reflection are minimized.

The effect of a "dephasing" wheel upon image quality was tested and found to be quite good in the case of the thin film domain observations. The dephasing wheel, placed in the incident laser light beam, reduces the coherence of the beam. Figures 1 and 2 (attached) are typical photographs of thin film domains made respectively without and with the dephasing wheel in use. The improvement in the image is apparent.

Attempts were made to observe domains on a (100) surface of the Fe-3% Si single crystal procured during Phase I of the program. As indicated in the Final Report for Phase I, we had already observed domains on this crystal by means of the powder pattern method. The attempts to observe such patterns magneto-optically have thus far been only partially

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successful. Most of the significant detail in the images is heavily masked by image noise which we have tentatively attributed to severe depolarization of the reflected beam by nonspecular scattering at imperfections in the crystal surface. Figure 3 is a photograph illustrating such a noisy image. By careful inspection of the photograph, alternate dark and light bands can be seen. We tentatively interpret this to be due to the underlying domain structure of the crystal. It is evident that the image noise must be greatly reduced before useful domain observations can be made. There are two approaches to such improvement. First, the optical quality of the crystal surface can possibly be improved by various polishing methods. Secondly, spatial filtering approaches to optical image enhancement can be applied. We plan to pursue both these approaches.

On June 30 and July 1, 1969, Dr. C. G. Gardner, Dr. D. L. Davidson, and Mr. J. R. Barton attended an informational meeting convened by Dr. O. Conrad Trulson of the Advanced Research Projects Agency. At this meeting Dr. Gardner reviewed the past and planned future work of the project in an oral and projection slide presentation.

The Phase I Final Report was prepared and submitted on 18 September.

Our plans for the coming quarter also include (1) a continuation of the magneto-optic study, with emphasis on overcoming the difficulties previously mentioned; (2) resumption of effort on the inductive coil method of detecting Barkhausen jumps; and (3) continuation of work on the theory of the Barkhausen effect. For the magneto-optic work, we plan to mechanically polish a surface of the single crystal, and vacuum anneal it afterwards, since our experience indicates that electropolishing will not lead to a surface of acceptable optical quality. In pursuing the induction coil method, we plan to work on the following principal tasks: (1) investigation of approaches to probe coil designs which would be selectively sensitive to Barkhausen jumps at specific depths within a specimen (a development which we think would reduce the dependence of the system response to specimen geometry); and (2) selection, preparation, and characterization of some pedigreed axial test specimens for use in explicit studies of the characteristics of Barkhausen Noise and magnetoabsorption as a function of applied stress.

If you should desire further information or clarification of the work reported here, we would be pleased to provide it.

Very truly yours,

Rufus H. Holloway
for John R. Barton, Director
Instrumentation Research

CGG:r

FIGURE 1. PHOTOGRAPH OF A REPRESENTATIVE DOMAIN PATTERN IN AN IRON THIN FILM SPECIMEN, MADE WITHOUT THE USE OF A DEPHASER IN THE INCIDENT LASER BEAM

FIGURE 2. SAME AS FIGURE 1, EXCEPT WITH DEPHASER IN INCIDENT BEAM

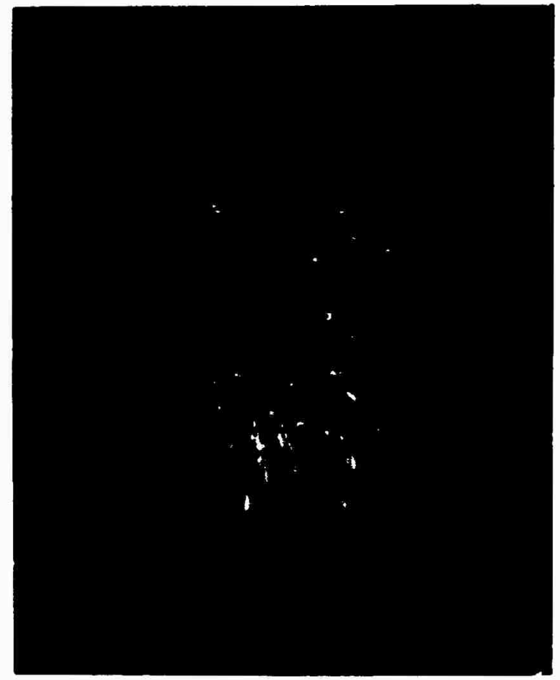

FIGURE 3. REPRESENTATIVE PHOTOGRAPH OF THE IMAGE OF THE (100) SURFACE OF AN Fe SINGLE CRYSTAL, MADE UNDER CIRCUMSTANCES COMPARABLE TO THOSE FOR FIGURE 2. THE DOMAIN PATTERN IS BARELY DISCERNIBLE THROUGH THE IMAGE NOISE

SOUTHWEST RESEARCH INSTITUTE

8500 CULEBRA ROAD SAN ANTONIO, TEXAS 78228

14 January 1970

Supply Officer
Naval Air Engineering Center
Philadelphia, Pennsylvania 19112

Attention: AS-63 for MAM

0856

Subject: Contract No. N00156-69-C-[redacted]; Second Quarterly Technical Progress Report, SwRI Project No. 15-2474, Phase II, "Development of Nondestructive Testing Techniques for Detecting Stress in Brittle Materials"

Dear Sir:

In summary, our activity during the past quarter of the project was as follows:

(1) An effort was made to improve the optical quality of the surface of our Fe-3%Si single crystal. This led to satisfactory magneto-optic domain images.

(2) Barkhausen jumps in permalloy thin films were visually observed and also photometrically recorded. A limited amount of data on jump size distribution was obtained, analyzed, and compared with existing theory.

(3) The general problem of high speed magneto-optic motion pictures of moving domain walls in the crystal while it is under mechanical stress was investigated.

(4) Some limited attempts at magneto-optic image enhancement by Fourier optical methods were made. Improvement was very marginal; however, our efforts were by no means definitive.

(5) Cylindrical specimens were fabricated for a pulse depth discrimination experiment. Further progress was made on the design of this experiment.

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As reported in our preceding Quarterly Report (dated 3 October 1969), the optical quality of the surface of our single crystal was observed to be poor even after careful polishing by standard metallographic methods. The poor quality was traced to extensive and very dense pitting of the surface. After extensive and careful polishing, followed by a twelve-hour vacuum anneal, the quality of the surface was found to be sufficiently improved to make possible the magneto-optic observations of surface domains. Figure 1 shows a typical photographic record of a domain pattern. (This may be compared with Figure 2 of our preceding quarterly report.) The contrast is quite good; there is still evidence of pitting of the surface. The coherent light speckle pattern, though greatly ameliorated by the dephasing wheel, is still discernible.

Over a period of several days, the crystal was observed to be tarnishing (despite the fact that it is stored in an air-tight dessicator when not in use). Upon repolishing and annealing, the optical quality of the surface was observed to have deteriorated badly. The reason for this is not definitely known; we have tentatively ascribed it to uncovering a shallow layer of voids or silicate inclusions. Efforts are currently being made to restore the surface quality and to establish definitely the reason for its deterioration, i.e., whether the fault lies in our polishing technique or in the material of the crystal itself, or both.*

As an exercise in preparation for similar work on the single crystal, a photomultiplier was introduced into the magneto-optic arrangement (at essentially the point where visual observations are made); its output was connected to a cathode ray oscilloscope, thus enabling us to record photometrically changes in the state of magnetization of some permalloy thin films. Figure 2 shows a hysteresis loop of such a film, made in the manner described. Note the clear manifestation of abrupt jumps in magnetization. Figure 3 shows an expanded oscilloscopic trace showing Barkhausen jumps of various magnitudes. Sufficient data of the type obtainable from Figure 3 was obtained to enable us to make a preliminary study of the Barkhausen jump size distribution in the permalloy film specimen. Figure 4 shows a graph of the so-called integral distribution in jump size, which is the number of observed jumps equal to or greater than a given size, versus jump size. The jump size is taken to be the ratio of the individual change in magnetization to the total change in magnetization from saturation to (directionally reversed) saturation. In the past, workers in this field have tried to fit such data to the graph of an

*Recent results indicate that the pitting is caused by the metallographic polishing procedure; a modified polishing procedure seems now to have eliminated the problem.

empirical relation of the form

$$N/N_o = \exp\left[-A\,(M/M_T)^{1/n}\right]$$

where N = number of jumps of size M or greater
N_o = total number of jumps (of all sizes)
A = an empirically determined constant
M = magnetization change in a given Barkhausen jump
M_T = total saturation magnetization of the specimen region in which magnetization changes are observed.
n = an empirically determined constant

Our data is roughly consistent with this relation for $n = 1$, but the agreement is not impressive.

Looking ahead to the task of domain dynamics studies, we have begun to analyze the motion picture problem. The high-speed camera available to us is capable of about 6000 frames per second. From a photometric analysis we have determined that our available laser sources do not have adequate luminous power to give an exposure at this speed. Consequently, we are examining the possibility of using either an incoherent source or a recently commercially introduced pulsed laser capable of high repetition rates.

The problem of magneto-optic image noise which we reported last quarter, and which we now know to be attributable to the previously mentioned surface pitting of the crystal, has been troublesome. We have made an attempt to improve the image quality by devising an optical "matched filter" to try to filter out of the image the light not due to the magneto-optic image itself. We were not successful with this. Image brightness suffered drastically, and little improvement in quality was discernible. However, we recognize that our technique for making and using the filter was unrefined, and better results might be obtainable with more effort. Since we found that we could (at least once) produce a satisfactory surface on the crystal, we dropped the image enhancement work.

Because we have been making good progress with the magneto-optic work, we have devoted less effort to the inductive coil experiments than we had originally projected. However, the latter aspect of the work has not been altogether neglected. We have in the past quarter carried out

a careful design study for experiments on cylindrical specimens. Some specimens have been fabricated at this writing, and a solenoid which can be used in conjunction with our stressing fixture has been designed. We expect all design and fabrication work to be complete in a few weeks, and that the planned experiments will be underway within the next quarter.

Our plans for the upcoming quarter are to continue the magneto-optic work, and especially to get it to the point where we can stress the specimen while we observe the domains. We are prepared to do this as soon as we can produce a satisfactory crystal surface that won't deteriorate before we can complete the experiment. In addition, we anticipate being able to complete the planned pulse depth discrimination experiment using the inductive coil approach.

Dr. George A. Matzkanin, a new member of the Southwest Research Institute staff, is now participating in the work of this project. For your information, we have attached a copy of his professional data sheet. Thus the project now has directly serving it Dr. C. G. Gardner as project manager, Dr. D. L. Davidson in charge of specimen design and preparation, and Dr. G. A. Matzkanin who is carrying out most of the laboratory work.

We will be happy to clarify or amplify any matter pertinent to the work briefly reported here should you wish to have us do so.

Very truly yours,

John R. Barton

John R. Barton, Director
Instrumentation Research

Prepared by:

C. G. Gardner

Encs.

Figure 1. Kerr magneto-optic image of magnetic domains on the surface of an Fe-3% Si single crystal.

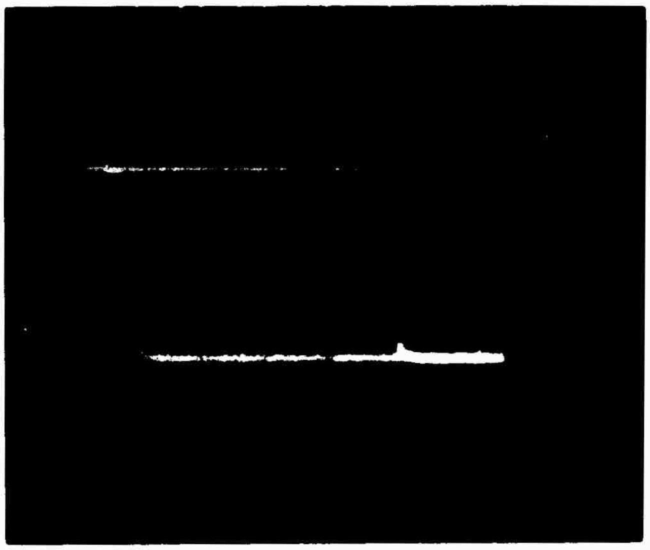

Figure 2. Magneto-optically determined hysteresis loop of a 77% Ni-23% Fe permalloy thin film (1000Å thick), obtained photometrically. The vertical axis is proportional to the magnetization of the specimen; the horizontal axis is proportional to the magnetic field applied to the specimen. Note the evidence of discontinuous changes (Barkhausen jumps) in the magnetization.

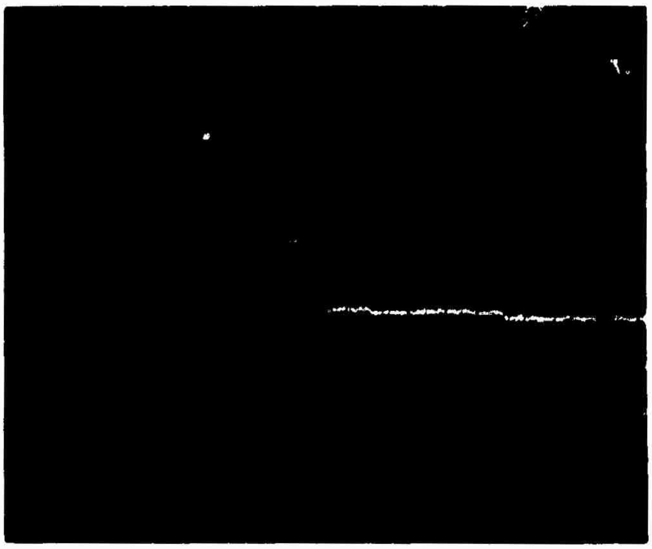

Figure 3. Magneto-optically and photometrically determined Barkhausen jumps in one complete reversal of magnetization of the permalloy thin film. Note the variation in magnitude of the jumps.

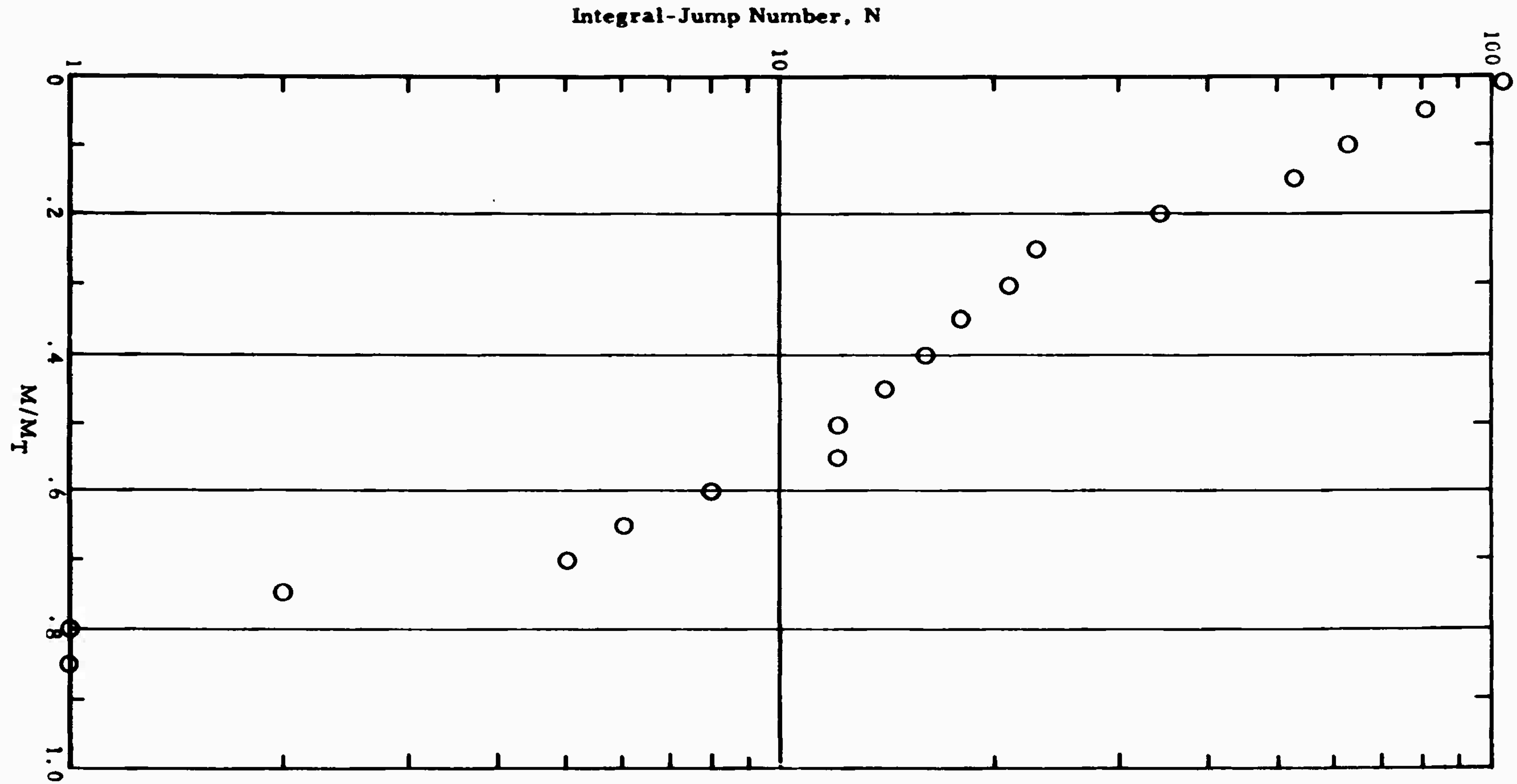

FIGURE 4. INTEGRAL SIZE DISTRIBUTION OF BARKHAUSEN JUMPS IN A 77% Ni-23% Fe PERMALLOY FILM 1600 Å THICK. JUMP SIGNS WERE MEASURED PHOTOMETRICALLY, USING THE KERR MAGNETO-OPTIC EFFECT

GEORGE A. MATZKANIN
Senior Research Physicist
Department of Instrumentation Research

A. B. in Physics, St. Mary's College (Winona, Minn.) 1960
M. S. in Physics, University of Florida, 1962
Ph. D. in Physics, University of Florida, 1966

An experimental physicist, Dr. Matzkanin's principal research experience includes applications of nuclear magnetic resonance and nuclear quadrupole resonance methods to studies of the solid state, and magneto-optic studies of the properties of ferromagnetic materials. In the course of his graduate work at the University of Florida, he employed nuclear magnetic resonance methods to investigate molecular motions and internal electric and magnetic fields in molecular solids, especially as these phenomena are influenced by high pressure and low temperature. Nuclear magnetic resonance studies of the Knight shift at high pressure led to noteworthy conclusions regarding the influence of lattice vibrations on the density of electronic states in lead and platinum, and the volume dependence of the anisotropic part of the conduction electron charge distribution in tin. Following his doctoral work, he moved to Argonne National Laboratory where he participated in studies of nuclear magnetic relaxation in metals and in alloys. In particular, he investigated the electronic structure in the neighborhood of solute atoms in dilute solid solutions, and also investigated magnetic coupling and the interaction of conduction electrons with localized magnetic moments in magnetic compounds of uranium. Since coming to Southwest Research Institute in 1969, Dr. Matzkanin has engaged primarily in investigations of the Barkhausen effect in ferromagnetics using the Kerr magneto-optic method as well as the standard inductive search coil method. He is the author or co-author of some eight scientific papers which have been published in the journals of professional societies.

PROFESSIONAL CHRONOLOGY: Teaching Assistant, University of Florida, 1960-2; Research Assistant, University of Florida, 1962-6; Postdoctoral Research Associate, Argonne National Laboratory, 1966-8; Visiting Assistant Professor of Physics, University of Illinois at Chicago Circle, 1968-9; Southwest Research Institute, 1969-(senior research physicist, department of instrumentation research, 1969-).

Memberships: American Physical Society, Society of the Sigma Xi.

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27 May 1970

Supply Officer
Naval Air Engineering Center
Philadelphia, Pennsylvania 19112

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Attention: AS-63 for MAM

Subject: Contract No. N00156-69-C-0856 Third Quarterly Technical Progress Report, SwRI Project No. 15-2474, Phase II, "Development of Nondestructive Testing Techniques for Detecting Stress in Brittle Materials"

Dear Sir:

A summary of our activities during the third quarter of the project is as follows:

(1) Modified polishing procedures making use of diamond grit resulted in a high-quality magneto-optic surface on our Fe-3% Si single crystal.

(2) Components were designed and fabricated for integrating the electromagnet into the stressing fixture so the single crystal specimen could be simultaneously stressed and magnetized while being observed magneto-optically.

(3) Static photographic data were taken of the effects of applied magnetic field and compressive stress on the domain patterns over the entire specimen surface.

(4) A normal-speed (16 fps) movie was successfully made of the domain dynamics involved in magnetization reversals of our single crystal.

(5) Preliminary results were obtained in using photometric techniques to sense Barkhausen jumps magneto-optically.

(6) The inductive coil Barkhausen experiments were initiated.

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Substantial progress was made in this past quarter on the magneto-optic effort as indicated by the above summary. Extensive static photographic data were taken and analyzed in terms of the effects of magnetic field and compressive stress on the magnetization process. The condensed results and interpretation are contained in a paper presented by us at the Washington Meeting of the American Physical Society, 27-30 April 1970. A copy of the text presented at the meeting is attached and constitutes the technical discussion section of this quarterly report.

During the next quarter, we plan to complete the analysis of the magneto-optic results at the present stage of the effort and write a paper on this work for publication by a refereed journal. We have obtained results in the course of this work pertinent to the general understanding of the effects of stress on magnetization processes and feel that publication is warranted at this time.

Although our magneto-optic work is currently refined to the point of providing much useful information on magnetization processes and domain dynamics, in order to stay within the framework of this year's project effort, we plan to spend the major portion of this coming quarter on the inductive coil Barkhausen experiments.

Should further information concerning the effort reported herein be desired, we shall of course be pleased to supply it.

Very truly yours,

John R. Barton

John R. Barton, Director
Instrumentation Research

Prepared by:

C. G. Gardner and
G. A. Matzkanin

Encs.

MAGNETO-OPTIC STUDY OF DOMAIN DYNAMICS
IN A SILICON-IRON SINGLE CRYSTAL

by

G. A. Matzkanin
D. L. Davidson
C. G. Gardner

Southwest Research Institute
San Antonio, Texas

Presented at the Washington Meeting of the American Physical Society, 27-30 April 1970

Work supported in part by the Advanced Research Projects Agency through the Naval Air Engineering Center, Philadelphia, Pennsylvania
ARPA Order No. 1247 Amend. 1
Program Code No. 9D10

In an effort to better understand the role of mechanical stress on magnetization processes in ferromagnetic materials, a program has been undertaken to study this problem from several standpoints. The work reported herein represents one aspect of this effort, namely, the study of magnetization processes by direct observation of magnetic domains. The Kerr magneto-optic effect has been used to study the static domain patterns on a (100) surface of a large single crystal of 3% Silicon-Iron which can be subjected to known mechanical loads. Discussed in this report are preliminary low-magnification photographic and visual studies of the effects of applied magnetic field and compressive stress on the overall static domain pattern.

Figure 1 shows schematically the basic experimental arrangement, which is essentially standard. A light beam, linearly polarized perpendicular to the plane of incidence, is reflected by the specimen and passed through a nearly-crossed polarization analyzer. Through the longitudinal Kerr effect, regions of localized magnetization on the specimen surface (magnetic domains) effect what amounts to a small rotation of the polarization vector, the sense of rotation depending on the sense of the surface magnetization of the specimen. A polarization analyzer set to extinguish rays whose polarization has been rotated in one sense will pass a small amount of light whose polarization has been rotated in the opposite sense. Thus, the image formed by the reflected beam will contain regions of differing brightness corresponding to the variation in magnetization direction among the domains.

The apparatus used incorporates a "C" electromagnet as an integral part of the stressing fixture through which both the field and stress can be varied. Figure 2 shows an overall view of the actual experimental arrangement. The stressing fixture is made of non-magnetic materials except for the magnet itself. Figure 3 is a closeup view of the magnet and specimen in the stressing fixture. The specimen is held in position by the magnet pole pieces.

The specimen is a single crystal of 3% Si-Fe approximately 3 cm long, 1 cm wide, and 3.5 mm thick. Careful mechanical polishing followed by a strain-relieving anneal under vacuum gave a satisfactory surface. The surface was not bloomed with a contrast-enhancing coating. Figure 4 shows schematically the specimen dimensions and orientation. The observed surface is nominally normal to a [100] direction with the other magnetically "easy" directions lying in the surface. Figure 5 shows some further details. On the left is a top view of the specimen surface. Note that the surface "easy" directions are not symmetrically oriented with respect to the specimen axis, but at angles of 55° and 35°, respectively. The specimen axis is 10° away from a $\langle 110 \rangle$ type direction. On the right, the crystal axes are shown projected on a plane normal to the reflected beam; this is the plane on which the magneto-optic image is formed.

Figure 6 shows the overall domain pattern observed on the surface of the demagnetized specimen. The largest domain widths are on the order of 3 mm, or about the same as the thickness of the specimen, suggesting that these domains extend through the crystal. The appearance of fir trees on this picture and on higher-magnification Bitter pattern pictures indicate the specimen surface is misoriented by about 1° with respect to a crystallographic (100) plane. Analysis of the pattern geometry indicates that the large V-shaped regions coincide with "easy" magnetization directions, whereas the walls most nearly parallel to the specimen axis are in a $\langle 110 \rangle$ direction. Because of the high anisotropy energy of Si-Fe, the magnetization is confined to "easy" $\langle 100 \rangle$ type directions, thus giving rise to 180° walls parallel to $\langle 100 \rangle$ directions, and 90° walls parallel to $\langle 110 \rangle$ directions. The arrows illustrate the assigned directions of surface magnetization. This interpretation is supported by a small but distinct contrast in image brightness which can be noted on careful inspection of the photographs. Although more evident in other higher-magnification photographs which have been made, these brightness variations can be seen in this picture in the "checkerboard" area. So instead of just "black" and "white," two distinct intermediate shades of gray are also observed. Analysis of the Kerr effect indicates that such contrast variations should be expected for the assigned magnetization directions. The four magnetization components parallel to the plane of incidence, to which the longitudinal Kerr effect is sensitive, differ in magnitude and direction, giving rise to the four domain shades observed in Figure 6. The relative brightness variations predicted from analysis of the Kerr effect for the particular specimen orientation

used in these experiments agrees with the interpretation of the magnetization directions given in Figure 6. The observation of such distinguishable intermediate brightness levels and corresponding contrast between domains has not previously been reported in the literature.

Figure 7 shows the effect of increasing the applied magnetic field along the specimen axis. The most favorably oriented domains (white) have increased in size, primarily by movement of 180° walls. Note the evident lower mobility of the 90° walls parallel to the ⟨110⟩ direction. Also note the beginnings of stripes in the other ⟨110⟩ direction. Figure 8 corresponds to a still greater applied field with the specimen almost completely taken over by the stripes. Visual examination of the dynamic magnetization process and the contrast variations of these stripes suggest that these are domains separated by 90° walls. The inequality of the width of alternate domains is interpreted as being due to the fact that the "easy" ⟨100⟩ directions are not symmetrically oriented with respect to the specimen axis, and unequal width is therefore expected in order to minimize the net transverse magnetization. As the field is further increased toward saturation, the less-favorably oriented domains (the darker, narrow ones) shrink and finally disappear.

The next several Figures show the effect of increasing the compressive load at zero applied field. First, Figure 9 shows the situation for a compressive stress of 900 psi; Figure 10 shows the pattern for 1700 psi. Note the appearance of more 180° domain walls in the "easy" direction farthest from the stress axis. Figure 11 gives the pattern for a stress of 2600 psi. Here the domains oriented in the "easy" direction at an angle of 55° with respect to the stress axis are dominant. At a stress of about 4000 psi, the demagnetized domain pattern consists entirely of these parallel, evenly spaced domains. This is a manifestation of the fact that, for positive magnetostriction materials, compressive stress forces the magnetization away from the stress axis. Comparison of the observed contrasts in this pattern with those noted in previous Figures tends to support this interpretation. If the field is now increased while the applied stress level is held fixed, observation shows that magnetization does <u>not</u> take place by simple movement of these 180° walls, but rather domains are nucleated in the other "easy" direction (the one at an angle of 35°) which then grow in the direction of the magnetizing field. This last point is currently being examined in greater detail and the magnetization process over the entire specimen surface is being analyzed at various levels of applied stress, both compressive and tensile.

Additional work has been done using photometric techniques for the study of discontinuous magnetization processes (Barkhausen jumps). The polarization-analyzed reflected beam can be monitored with a photomultiplier and oscilloscope while slowly sweeping the applied field. Abrupt changes

in magnetization can then be displayed as spikes on the scope by use of appropriate high-pass filters. Although the work is still in a preliminary stage, initial results seem to correlate well with results obtained by conventional inductive coil methods. Future plans include pursuing the photometric method of sensing surface Barkhausen jumps and studying domain dynamics by means of high-speed cinematography.

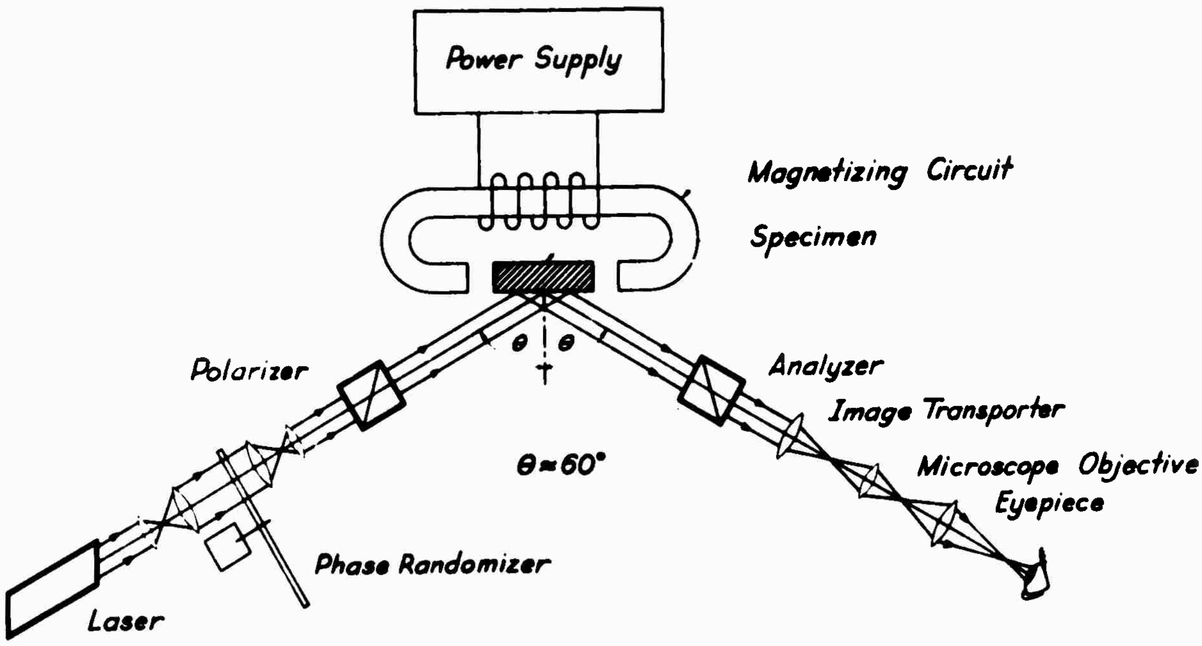


FIGURE 1. LONGITUDINAL KERR MAGNETO-OPTIC EFFECT DOMAIN OBSERVATION SYSTEM (SCHEMATIC)

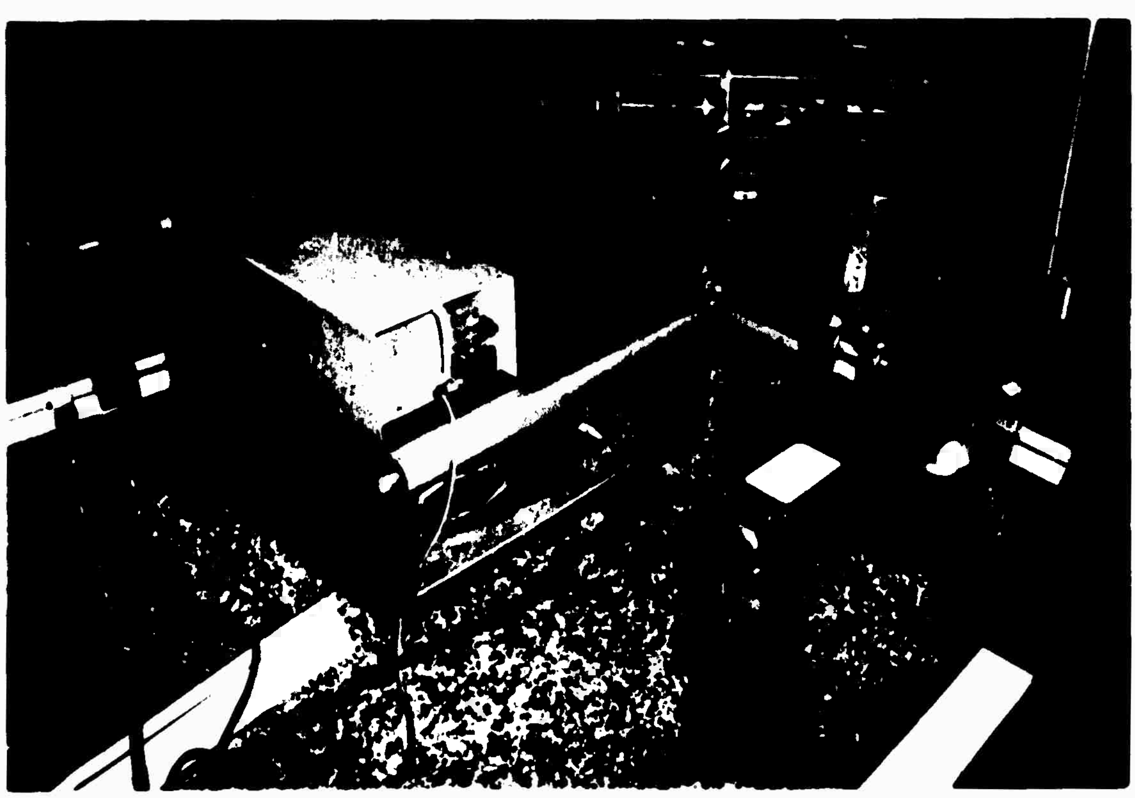


FIGURE 2. OVERALL VIEW OF EXPERIMENTAL ARRANGEMENT

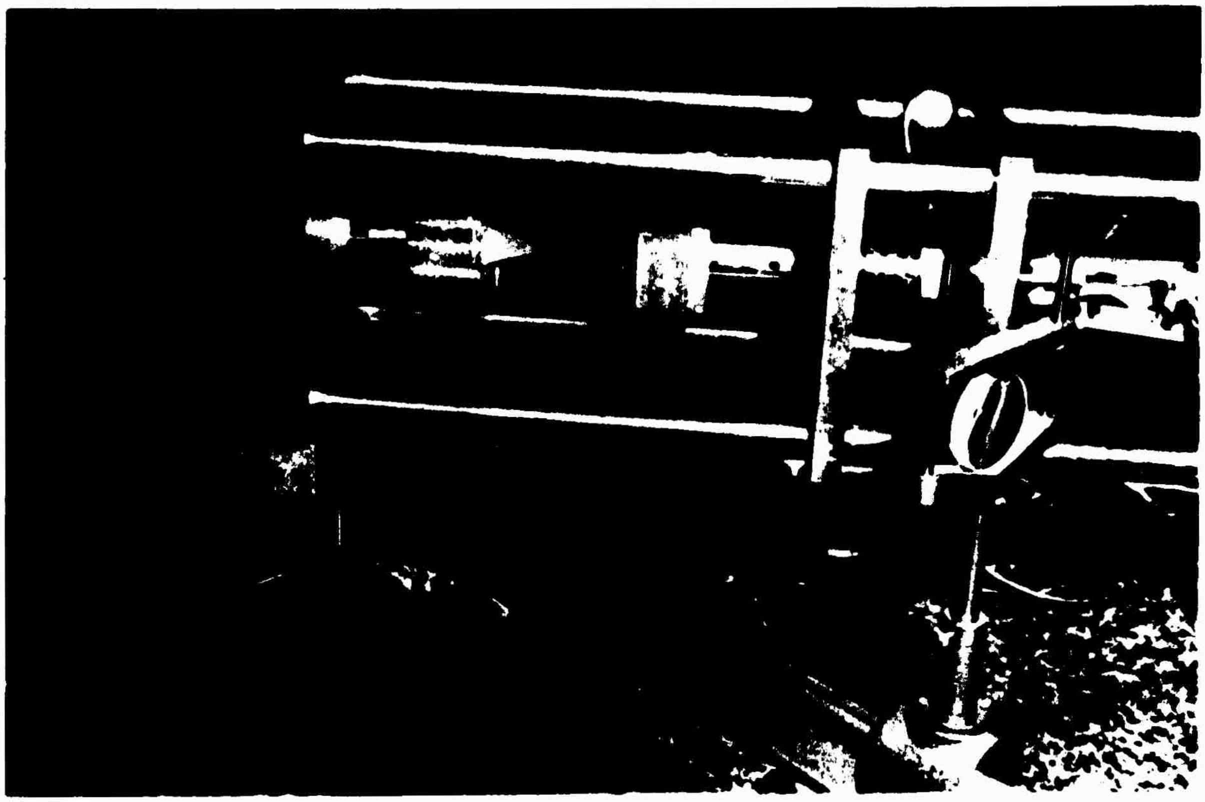


FIGURE 3. MAGNET AND SPECIMEN IN STRESSING FIXTURE

RECTANGULAR Fe-3% Si SINGLE CRYSTAL SPECIMEN

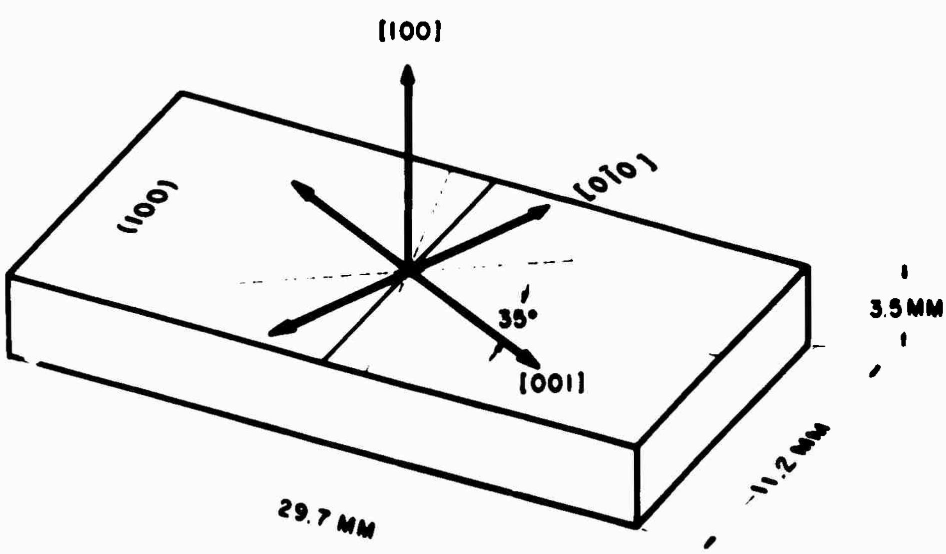
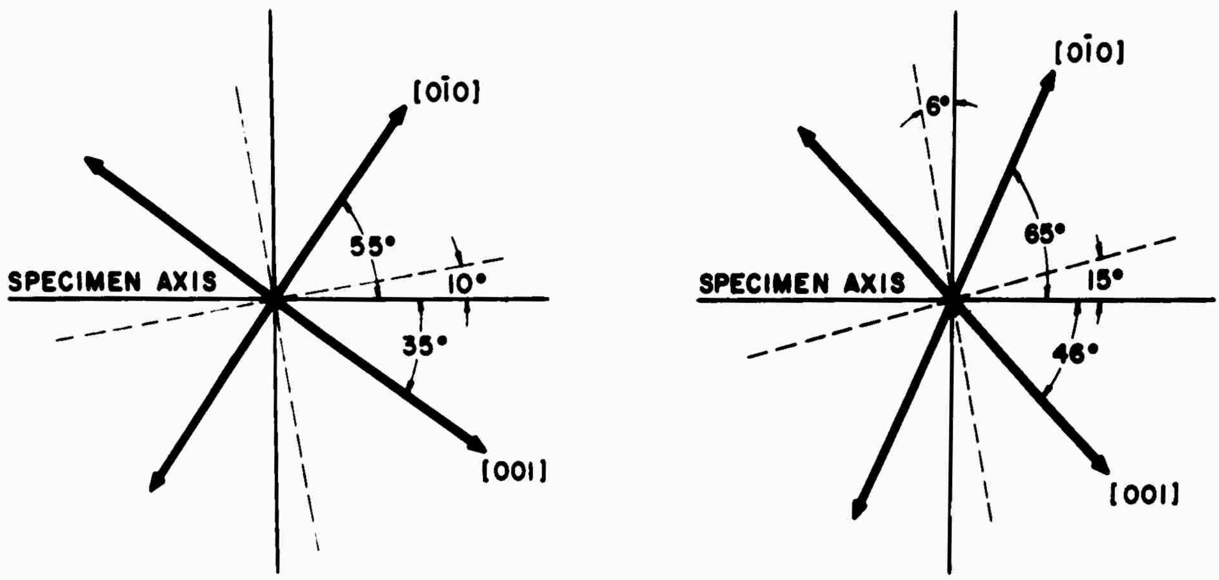


FIGURE 4. SPECIMEN DIMENSIONS AND ORIENTATION



TOP VIEW OF SPECIMEN (100) SURFACE.

PROJECTION OF CRYSTAL AXES ON A PLANE NORMAL TO THE REFLECTED BEAM. ANGLE OF INCIDENCE = 47.5°

FIGURE 5

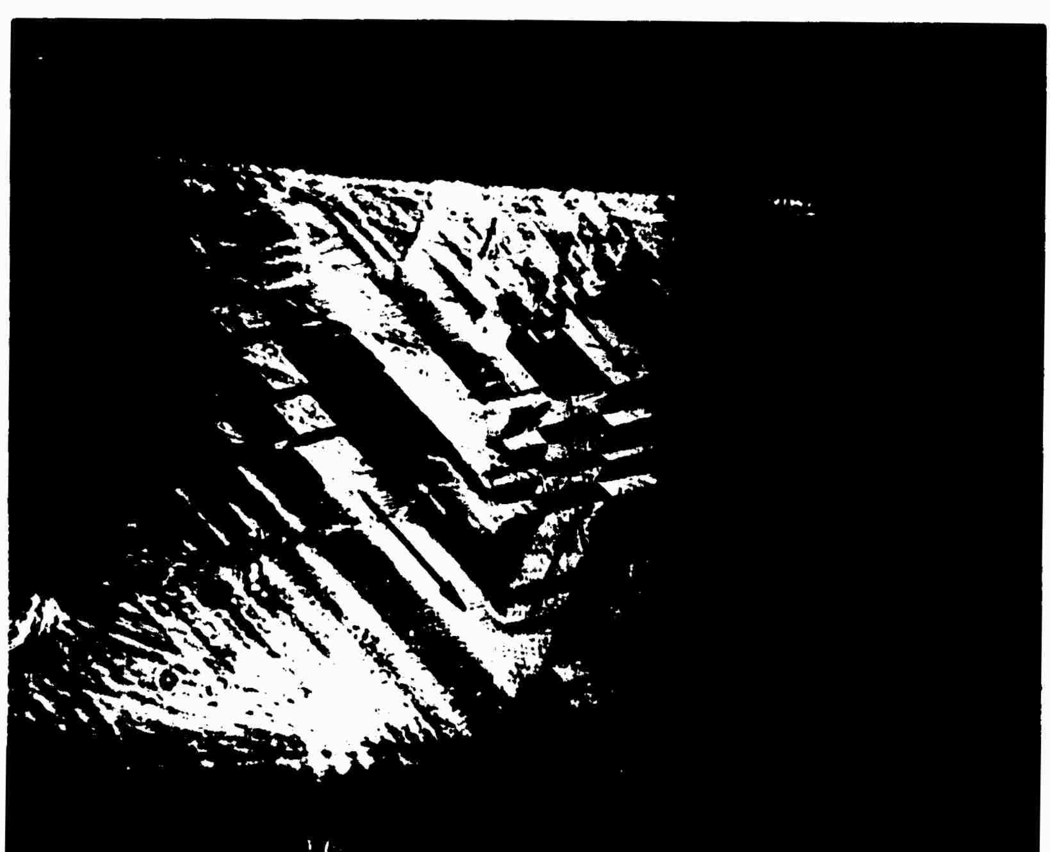


FIGURE 6. DEMAGNETIZED DOMAIN PATTERN

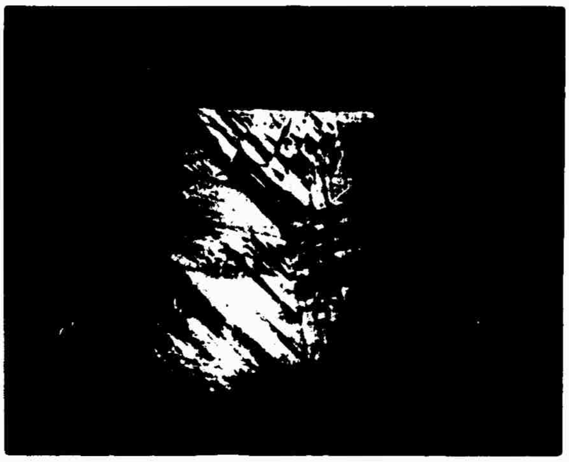


FIGURE 7. DOMAIN PATTERN WITH SMALL MAGNETIC FIELD APPLIED ALONG SPECIMEN AXIS

FIGURE 8. DOMAIN PATTERN AND 90° WALLS NEAR SATURATION

FIGURE 9. DOMAIN PATTERN WITH LONGITUDINAL COMPRESSIVE STRESS OF 900 PSI

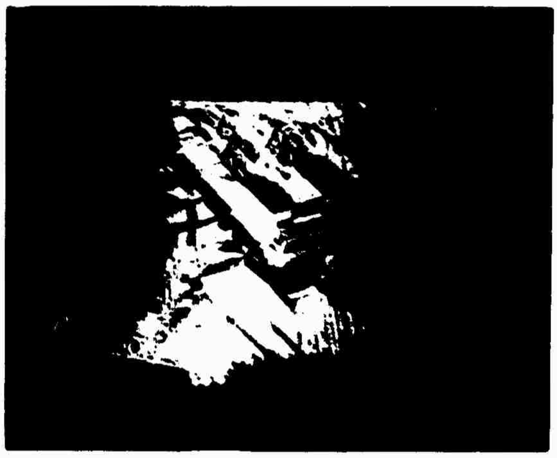


FIGURE 10. DOMAIN PATTERN WITH LONGITUDINAL COMPRESSIVE STRESS OF 1700 PSI

FIGURE 11. DOMAIN PATTERN WITH LONGITUDINAL COMPRESSIVE STRESS OF 2600 PSI